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USE OF CHEMICAL ADDITIVES TO IMPROVE AERATION RATES. STUDY OF THE BEHAVIOR OF SINGLE BUBBLES

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Maine University
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1966

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Progress Report 2a

Use of Chemical Additives To Improve Aeration Rates

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Study of the Behavior of Single Bubbles

by
STEFAN A. ZIEMINSKI
and
DELMAR R. RAYMOND

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U. S. Department
of
Health, Education, and Welfare

PUBLIC HEALTH SERVICE
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PROGRESS REPORT NO. IIa

USE OF CHEMICAL ADDITIVES TO IMPROVE AERATION RATES

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STUDY OF THE BEHAVIOR OF SINGLE BUBBLES

Details of illustrations in this document may be better studied on microfiche.

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STEFAN A. ZIEMINSKI

AND

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U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

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ABSTRACT

This report deals with construction and testing of equipment for the study of the behavior of a single bubble in the presence of minute quantities of organic substances. The variables proposed for investigation are the velocity of rise, instantaneous dissolution rate, shape, oscillation and effects of age of the bubble on the mass transfer.

One of the main objectives of the design of the equipment was to obtain a continuous record of the behavior of the bubble from the moment of its formation to the end of its journey at the top of the column.

The principle used to achieve this objective was to follow by means of high speed photography the motion of the bubble simultaneously with the changes of its volume as indicated by the position of the meniscus in the capillary of a dilatometer.

INTRODUCTION

The work presented in our previous report (7) was concerned primarily with the behavior of a swarm of air bubbles in water in the presence of minute quantities of some organic substances. The substances selected for that study were some members of the homologous series of mono- and di-carboxylic acids and aliphatic alcohols. The results of this investigation showed that a considerable increase in the rate of oxygen transfer can be achieved by small amounts of some of these substances; e.g., heptanoic acid at a concentration of 10 ppm increased the rate of oxygen transfer by over 100 per cent as compared with that in pure water. It was also found that the magnitude of this effect depends on the structure and molecular size of the organic compound.

All these tests were conducted with a swarm of air bubbles produced by a porous plate. This was done because it was realized that coalescence plays here an important role and, in addition, a system of this kind offers the advantage of obtaining data more closely related to the actual aeration practice.

The disadvantage of this system is the complication caused by the interaction of the bubbles and the difficulty in the rigorous treatment of the results.

It was, therefore, decided to continue the above investigation with the use of a single-bubble system and to evaluate the effects of the agents on quantities such as the velocity of rise, instantaneous dissolution rate, shape and oscillation of the bubble. The effect of the age of the bubble on its solution rate was also proposed for investigation.

The main difficulty in a study of this kind is the construction of the experimental equipment and development of a technique which will give sufficiently accurate and reproducible results.

The main objective of our present work was to build and test the accuracy of a single-bubble equipment, using the dilatometric technique and high speed photography for the measurement of the instantaneous volume changes of the bubble.

REVIEW OF THE PREVIOUS WORK

The dilatometric technique was successfully applied by Ledig and Weaver (4) and Ledig (5) in the study of absorption rates of a carbon dioxide bubble in a sodium hydroxide solution. They used a closed system open to air only through a capillary tube. The resulting contraction of the bubble produced motion in the thread of solution in the capillary which was followed by means of shadowgraphs. Recently Leonard and Houghton (6) have applied this principle in the study of mass transfer phenomena for single bubbles. In their work the capillary was filled with mercury and the motion of the mercury thread was recorded by means of a platinum resistance wire stretched along the capillary.

Probably one of the more important factors affecting the accuracy of this method is the sensitivity of the response of the capillary fluid to the changes in volume of the dissolving bubble. The higher the rate of absorption the greater is the possible error produced by the delay in response. Generally, the capillary liquid should have a low density and viscosity and the diameter of the capillary should be consistent with the rate of volume change to avoid excessive lag in response.

The use of mercury should be avoided, and whenever possible the absorbing solution itself should be used as the capillary liquid.

Hammerton and Garner (3) in their studies of absorption from single bubbles have also used a dilatometric technique with water as the indicating liquid. In this case a photographic record of the movement of the meniscus in the capillary was used to determine the actual volume of the bubble at any desired time. According to the above authors the accuracy of their method was 1.2×10^{-4} ml. Unfortunately, however, the brief description of their method does not allow conclusions to be drawn regarding possible sources of error.

The successful application of the dilatometric technique requires a rigid air-tight system completely filled with the absorbing liquid, with the capillary as the only connection with the atmosphere. This requirement calls for a special design of the bubble release arrangement, which should maintain the closed system after release of the bubble. The release system which satisfies the above condition is the rotary bubble cup (1,6). Here the bubble is injected under the bubble cup and released by inverting it. This arrangement, although satisfying the requirement of a closed system, has the obvious disadvantage of introducing turbulence, excessive oscillation of the bubble, etc., which could affect the accuracy and reproducibility of the results. It constitutes also a potential source of leakage from the absorption column which in the case of smaller bubbles may lead to serious errors.

Another requirement of the dilatometric method is the constancy of the volume of the experimental equipment. This is a very important requirement since very small volume changes are to be measured.

The principal factors affecting the volume of the equipment are the temperature changes and mechanical factors responsible for stresses in some parts of the equipment resulting in changes of shape and volume. Despite the fact that the duration of the test is usually short (a few seconds) the above factors should be considered in the design of the equipment and if possible necessary corrections introduced.

THE EXPERIMENTAL EQUIPMENT

One of the main objectives of the design of the absorption equipment was to obtain an accurate and continuous record of the behavior of the bubble from the moment of release to the end of its journey at the top of the column. The principle used to achieve this objective was to follow, by means of high speed photography, the motion of the bubble simultaneously with the changes in volume as indicated by the capillary liquid. At camera speeds of about 1000 frames per second an almost continuous record can be obtained of the bubble size, shape, oscillation, etc., as related to the height of the column and the time elapsed from the moment of the bubble release.

(a) General Description (See Figures 1 and 2)

Water redistilled in a borosilicate glass system (Barnstead Redistiller) was introduced into a plexiglas stripping column where it was stripped from the dissolved gases by means of helium under vacuum. After the stripping operation the water was transferred under moderate vacuum into the absorption column. Special care must be taken to avoid entrapping of gas bubbles. After the column had been filled it was left for a time necessary

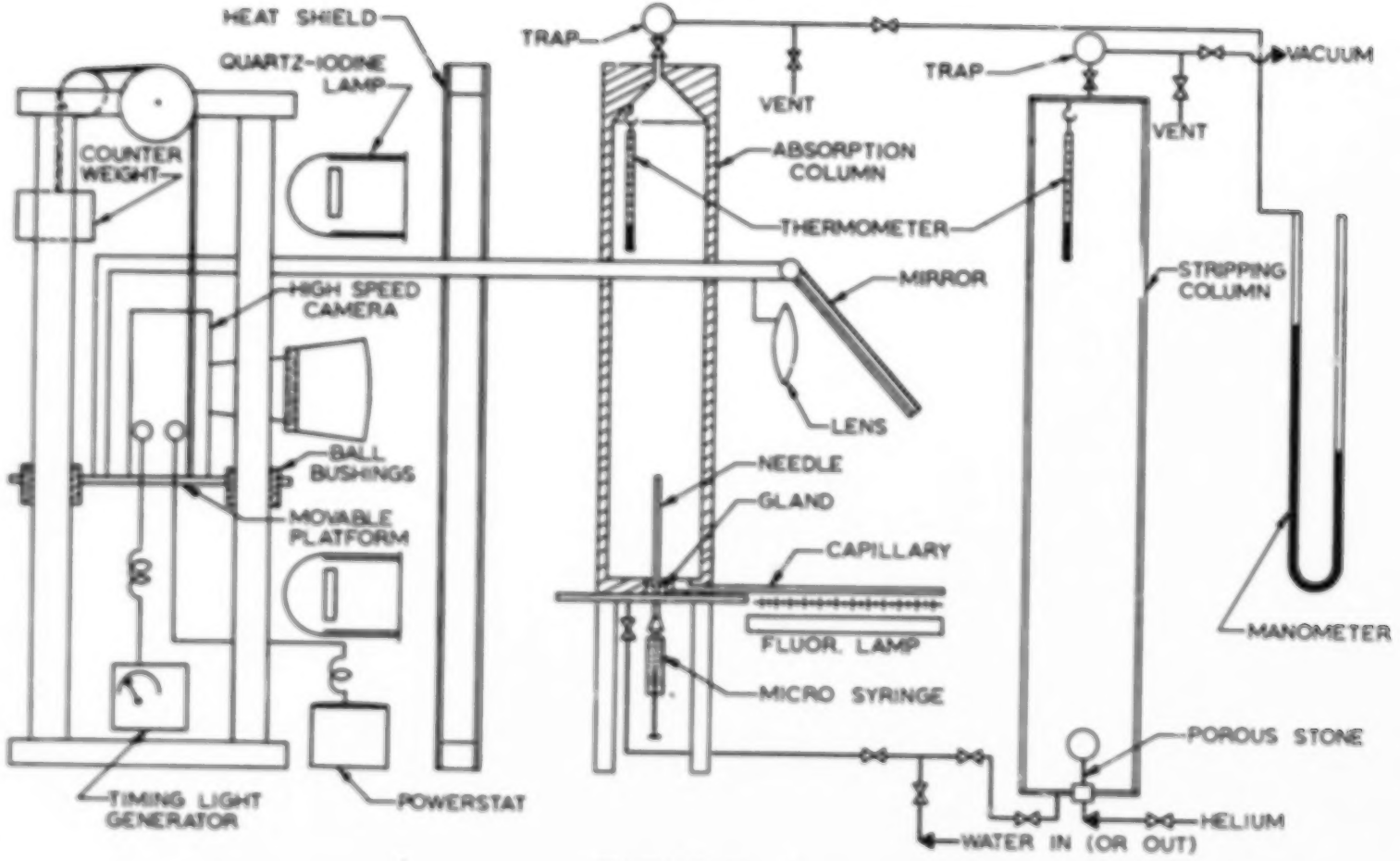

FIGURE 1
DIAGRAM OF THE EXPERIMENTAL EQUIPMENT

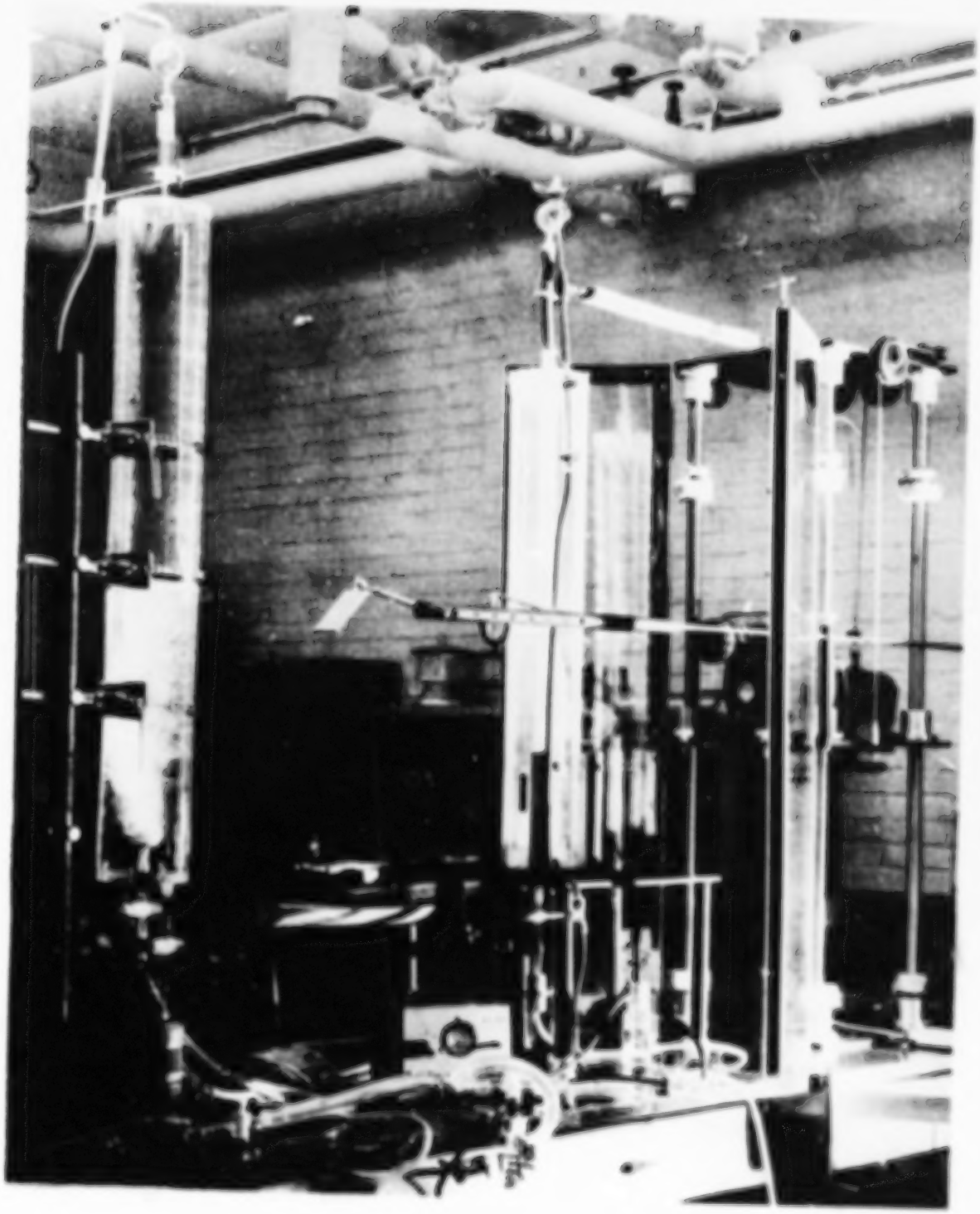

Figure 2.

Photograph of the Equipment

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to allow the water to adjust itself to the temperature of 25°C at which the absorption test was conducted.

The gas bubble was introduced at the bottom of the column by means of a hypodermic needle and a micro-syringe. Before the release of the bubble the meniscus of water in the capillary was set at the zero mark. One or two seconds before the release, the lights and the camera were switched on.

The camera was mounted on a well-balanced movable platform so that the process of the rise of the bubble could be accurately followed by hand control.

The film was developed in the Cramer Continuous Film Processor and was ready for analysis in about 30 minutes after completion of the test.

The film was analyzed by means of the Bell & Howell (Diplomat 16 mm) projector. The projector is equipped with a very convenient frame counter which can be reset at any point and which subtracts when the projector is run in reverse.

(b) Detailed Description

The Absorption Column: The column was made of 1/2 inch plexiglas plates. It is 82 cm high and has an inside cross section of 6 cm by 6 cm. It can be seen from Figure 3 that the base of the column is provided with a small well connected to a capillary tube. In order to increase the speed of response of the capillary liquid the length of the capillary was kept at a minimum corresponding to the greatest change in volume of the bubble. The connecting tube joining the well with the capillary has a length necessary to satisfy the optical requirements of the system, principally to give a sharp picture of the capillary meniscus at different elevations of the camera. The capillary was tested for uniformity of cross section by means

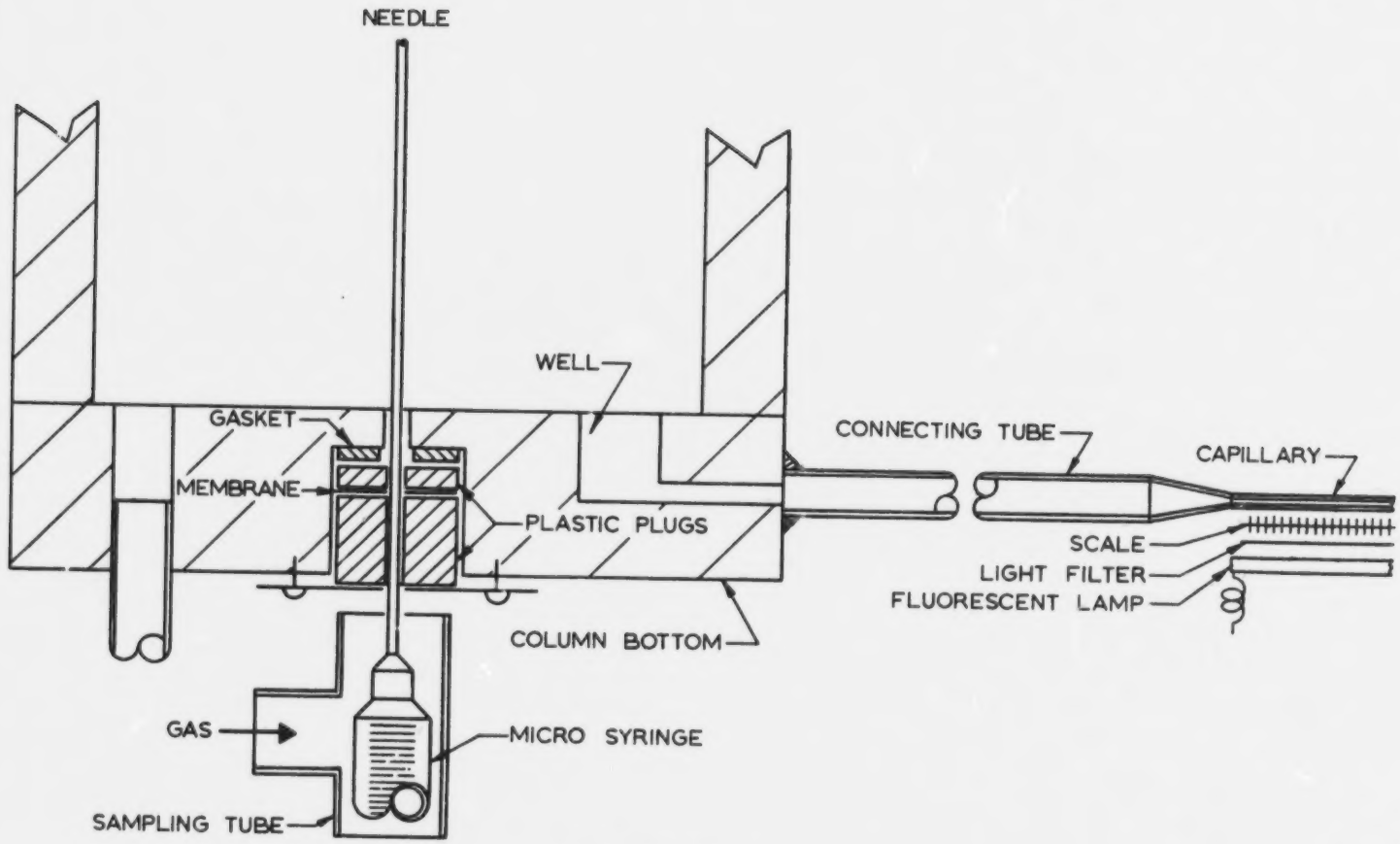

FIGURE 3
BUBBLE RELEASE ARRANGEMENT
(REMOVABLE NEEDLE)

of mercury. The cross section was found to be 0.006635 cm^2 and uniform throughout the length of the capillary. The transparent scale was located at the mid plane of the capillary to avoid a parallax error. The scale was illuminated by a fluorescent tubular lamp provided with a red filter. After each test the capillary was removed and left overnight in a cleaning solution.

Located in the center of the base plate is the arrangement for injection of the bubble (Figure 3). It consists of two machined plexiglas cylinders 1.58 cm in diameter fitting into a corresponding opening in the bottom plate of the column. At the top of the upper cylinder is a rubber gasket which seals it at its outer edge. A soft rubber membrane is placed between the cylinders. The cylinders are then pulled tightly together by means of a yoke located at the bottom of the base plate.

Both the plastic cylinders have a central opening of a diameter slightly larger than that of the hypodermic needle used for release of the bubble.

In most of our helium experiments a 713 gauge hypodermic needle was used. The needle was attached to the Hamilton gas-tight-syringe, filled with gas and inserted in the column through the cylinder opening. The punctured rubber membrane formed an excellent seal between the needle and the base of the column.

To fill and drain the column the base plate was provided with a teflon-plug stop-cock which served also for the adjustment of the position of the capillary meniscus after introduction of the needle.

The top of the column was made of a 3-1/2 cm plexiglas plate machined and polished to a conical shape. This conical top simplifies the filling operation and allows removal of all the gas from the column. A thermometer is mounted inside the column.

(c) H. S. Camera and the Moving Platform

The Hycam high speed motion picture camera (Model K 1001) was used in photographing the bubbles. It is a 16 mm camera utilizing a rotating prism for optical compensation. It has a film capacity of 100 ft of standard thickness film. The frame rate is variable from 100 to 8500 pictures per second. The frame rate is changed by varying the input voltage (AC or DC) to the camera motor. A Superior's Powerstat Model 216, operating from 115V AC input was used for voltage regulation. It is satisfactory for frame rates of 500 to 6000 frames per second. Due to the characteristics of the Universal AC-DC motor, below 500 frames per second direct current must be used.

The Milli-Mite timing light generator was connected to the camera (Fig. 1). It is a portable battery-operated transistorized unit designed to put a 10, 100 or 1000 cycle time base on the moving film. The exact speed of the film can then be determined by means of marks exposed on the film by a neon timing light of the camera. The frequency of the timer was periodically checked by means of an electronic counter.

The camera was mounted on a movable platform made of a 5/8" steel plate (Fig. 1). The platform was provided, at its corners, with four ball bushings moving on vertical, machined, steel shafts. The platform including the camera was suspended on a system of pulleys and balanced by means of a counterweight.

The use of ball bushings and machined shafts decreased the friction so that the platform could be easily moved by hand to follow the motion of the bubble in the column. This construction also allowed a uniform motion of the camera.

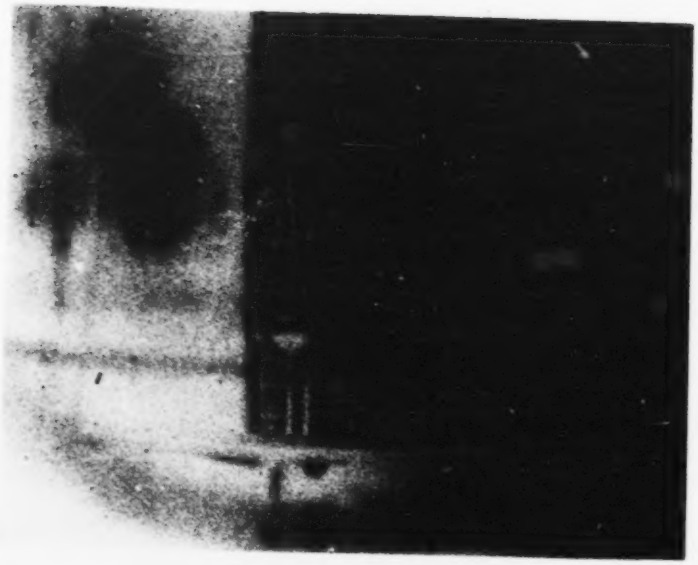

Figure 4

The Bubble and Its Capillary Reading

Attached to the platform is a horizontal bar carrying at its end a lens and a mirror. The horizontal axis of the lens and mirror system coincides with that of the camera lens and the mirror is directly above the capillary. This arrangement makes it possible to obtain pictures of the bubble simultaneously with pictures of the corresponding positions of the meniscus in the capillary.

Figure 4 shows an enlarged picture of one frame of the film. The Kodak Tri-X negative film was used in all the tests.

Illumination was provided by two Colortran quartz-iodine lights rated at 750 watts each. These lights (Fig. 1) were placed 100 cm from the column

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and at an angle of approximately 45° on each side of the column. Between the lights and the column are two heat shields, each constructed of two sheets of plexiglas with a dead space of air 2 cm thick. These heat shields help in decreasing the thermal expansion of the column.

OPERATION OF THE EQUIPMENT

Since the purpose of the equipment is to investigate effects of minute quantities of solutes on the behavior of gas bubbles in water, the purity of the water used in the tests is of basic importance. All the tests were conducted with distilled water, redistilled in a Barnstead (borosilicate) redistiller. To remove dissolved gases the water was introduced into the stripping column (Fig. 1) and stripped with helium under vacuum. Before the column was filled, the temperature of the water was adjusted between 23.5 and 26.5°C depending on the room temperature. If the room temperature was higher than the test temperature of 25°C, the lower water temperature was used and after filling of the absorption column, it was left there until the required temperature was reached. The stripping column was filled to approximately 2/3 of its volume which is about 3-1/2 liters. The actual stripping operation was conducted at 20 inch of Hg vacuum for 5 minutes.

After stripping the water with helium the absorption column was filled. A rubber cap was placed at the end of the capillary to prevent leakage of air, the column was rinsed with helium and brought under moderate vacuum. The vacuum above the water level in the stripper was decreased by means of helium and the water slowly transferred to the absorption column. Extreme care was taken to avoid entrapment of gas bubbles or their attachment to the

wall of the column. When the water appeared in the trap the valve at the top of the column was closed , the vacuum was released from the vacuum conduit, and the cap removed from the end of the capillary.

(a) Adjustment of the Lights and Camera

It was found that 2 quartz-iodine 750 watt lights are sufficient for illumination. This intensity of illumination and the arrangements of the lights previously discussed are correct only for the speed of about 1000 frames per second and when the camera, mirror, lens and column are in an exact relative position. To change any of these factors would require a corresponding change in the lighting arrangement.

Another important point in the illumination of the event is the adjustment of the fluorescent lamp under the capillary scale.

Once the lighting has been adjusted the camera must be focused. This focusing must be done on both the center of the column (path of the bubble) and the lens-mirror arrangement. From trial and error procedure it was found that the correct relative positioning of the components is: camera to column 20.5 inches, column to lens 2.5 inches, and lens to mirror top 12.0 inches. These measurements must be observed to 1/8" if a good, clear picture is to be obtained. When these preliminary adjustments have been completed the actual focusing is done through the camera. The camera is moved on the platform to a height of 24 inches above the capillary scale and focused on the capillary. The distance of 24 inches was found to be the best focusing position.

(b) The Bubble Release

When the water in the column has reached the test temperature of 25°C the syringe with the attached needle is filled with water up to the 0.004 ml

mark. At this setting the drop of water at the tip of the needle is removed to obtain a level water surface.

In the case of helium tests, helium is passed into the side arm of a glass "T" tube (see Fig. 1) located directly under the needle entrance opening in the base plate of the column. The needle is then introduced into the vertical part of the "T" tube and filled with gas to the mark corresponding to the desired bubble volume plus 0.004 ml. The excess of 0.004 ml of water is discharged into the column (when the plunger reaches the top of syringe) and thus cuts off the bubble from the needle tip.

When the syringe is filled with the desired volume of gas it is inserted into the column by puncturing the gland membrane. This method was used in helium tests for checking accuracy and reproducibility of the equipment. It have proved very satisfactory in this respect. However, it was found that in the case of easily soluble gases and under certain operating conditions this method of bubble release may result in errors in the volume determination. This point as well as another method for bubble release are discussed in a later part of this report.

When the needle is inserted inside the column the meniscus of water in the capillary is adjusted to the zero mark. Since the meniscus responds to very small changes of temperature and pressure the remaining phases of operation must be done quickly and carefully. The camera is put at its lower position facing the tip of the hypodermic needle. The lights and the camera switch are then turned on. The platform is held stationary for a fraction of a second while the camera reaches a constant speed, following which the bubble is released by depressing the plunger of the syringe and the camera is moved upwards with the velocity of the rising bubble.

Immediately after the run is completed the temperature correction of the capillary reading is made. This is done by noting the displacement of the capillary meniscus during 5 seconds with the photographic lamps on. The correction is expressed in millimeters of capillary displacement per second. In most cases it did not exceed 2% of the bubble volume. Another correction for the capillary reading is that for the layer of water adhering to the capillary wall. This correction is estimated by displacing the layer by means of mercury and measuring its volume. After completion of the test the film is processed in the Cramer continuous film processor and in about 50 minutes is ready for analysis.

Two methods of analysis were used for evaluation of the size, position and other characteristics of the bubble. In one method the film was projected on the screen by a Bell & Howell 16 mm projector and the projected measurements of the bubble and the needle were taken by means of a divider. Since the diameter of the needle was known, the actual bubble measurements could be easily calculated. The position of the bubble with respect to time was determined from the known speed of the film, frame counter and graduation marks on the column.

In the other method the lens system of the projector was removed and substituted by a 20 power microscope provided with a micrometer eyepiece. This method has proven to be faster and more convenient than the first one.

ACCURACY OF THE EXPERIMENTAL PROCEDURE

To check the accuracy and reproducibility of the technique a series of tests were conducted with helium bubbles. Helium is especially suitable for this purpose because of its low solubility in water. The volume change of the rising bubble under isothermal conditions, as calculated from the gas law, can be compared with that experimentally determined from the displacement of the meniscus in the capillary. Figure 5 shows the results of a series of helium tests conducted with bubbles of a volume of 0.016, 0.026, and 0.034 cc. It shows an excellent agreement between the experimental data as indicated by the run symbols and the continuous line representing the calculated values.

Figure 6 shows the volume changes with respect to time. The time zero on the abscissa of the graph marks the moment at which the bubble starts emerging from the tip of the needle. The steep portion of each curve includes therefore the time of bubble formation and most of the delay in response which is greatest during the growth of the bubble. Since the time of bubble formation varied between 0.15 and 0.25 of a second the delay of response, as approximated from the graph, was about 0.4 sec and independent of the bubble size.

It is of interest to note that in this group of tests the capillary thread was about 40 cm long. In our subsequent tests the length of the capillary thread was shortened to about 15 cm by inserting a larger connecting tube (see Figure 3). This decreased the frictional resistance and improved the response of the capillary fluid considerably. To obtain good response it is of importance to have the capillary tube as short as possible.

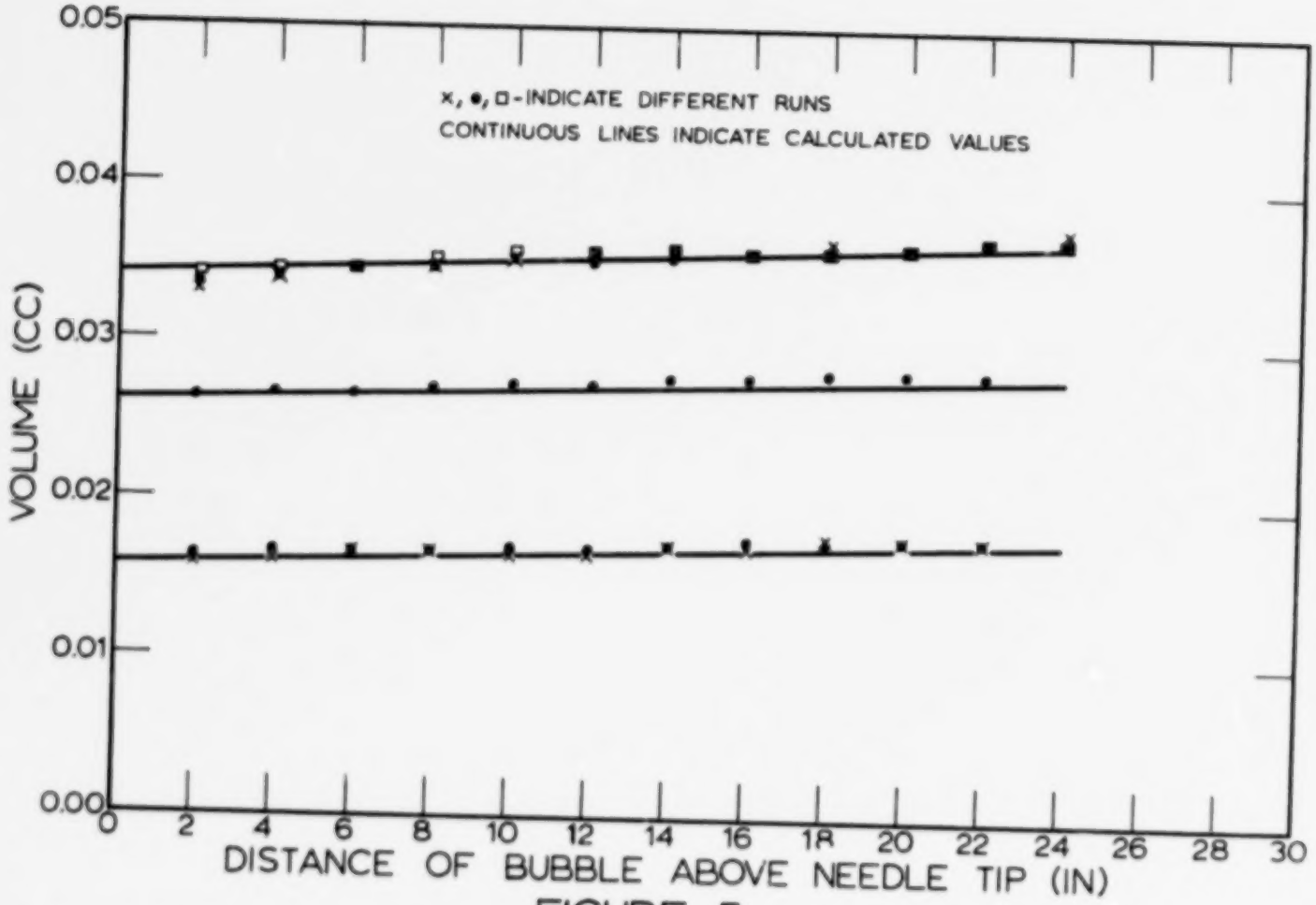

FIGURE 5

VOLUME OF BUBBLE vs COLUMN HEIGHT - HELIUM TESTS

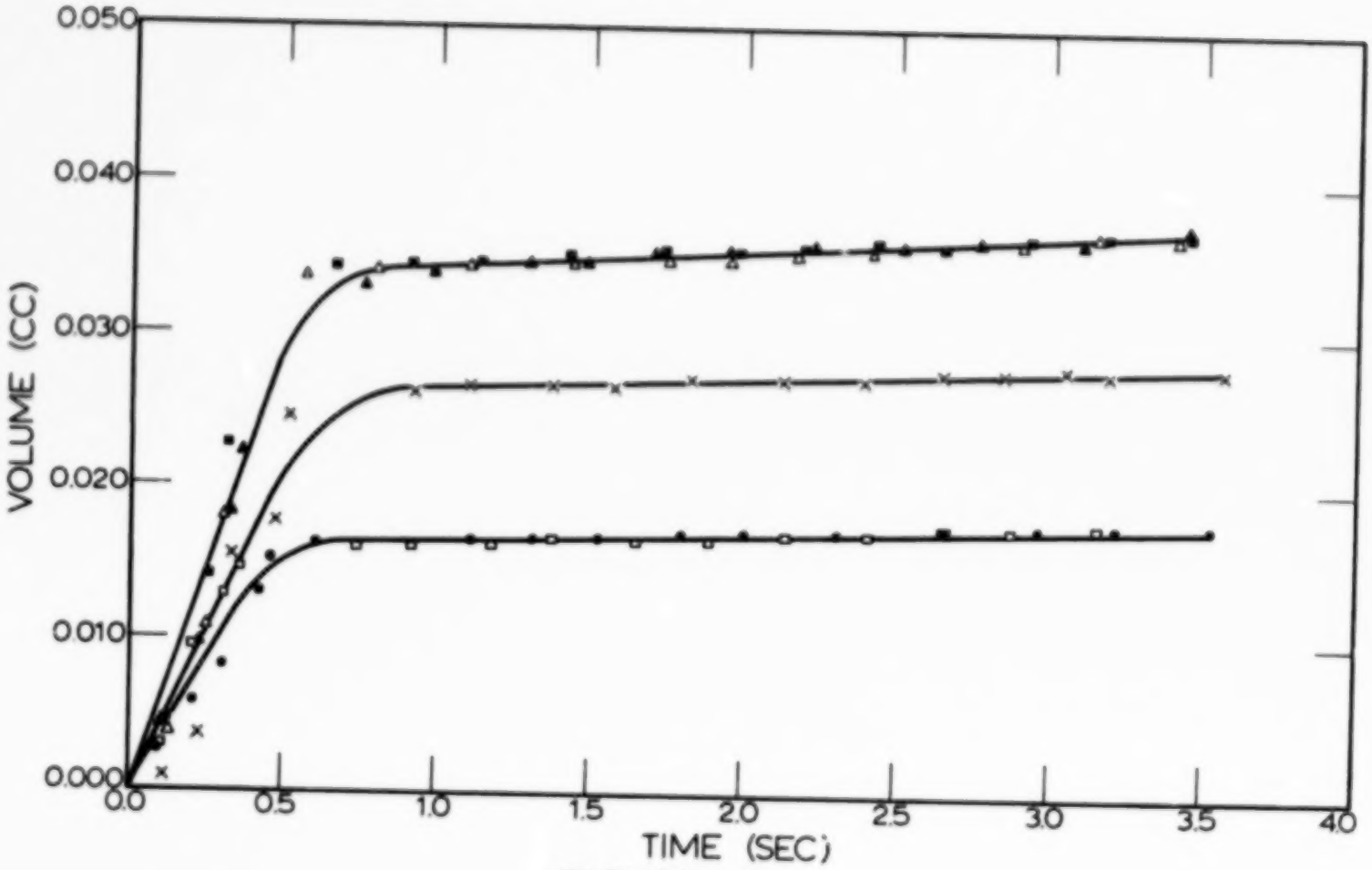

FIGURE 6
VOLUME OF BUBBLE vs TIME - HELIUM TESTS

CALCULATION PROCEDURE

The rate equation for mass transfer from a bubble to the surrounding liquid is given by:

$$-\frac{dn}{dt} = K_L A(C_o - C_L) \qquad (1)$$

and the volume pressure relationship of the rising bubble at any given time can be expressed by:

$$PV = nRT \qquad (2)$$

Substituting eq. (2) into eq. (1) and assuming isothermal conditions of the tests results:

$$-\frac{1}{RT}\frac{d}{dt}(PV) = K_L A(C_o - C_L) \qquad (3)$$

Since the liquid is originally free from the gas being absorbed, C_L may be taken as equal to zero and for the case where Henry's Law holds the equilibrium concentration C_o can be expressed by:

$$C_o = HP \qquad (4)$$

The pressure at any point of the column is:

$$P = P_B - hc' \qquad (5)$$

where P_B is the barometric pressure, h is the distance measured from the bottom of the column to the bubble and c' a conversion factor.

Solving of eq. (3) and substituting eq. (4) and (5) yields:

$$K_L = \frac{1}{RTAH}\left[\frac{Vc'}{P_B - hc}\frac{dh}{dt} - \frac{dV}{dt}\right] \qquad (6)$$

The values of the derivatives can be easily determined from the slopes of the corresponding curves and the values of V are known from the capillary readings. The greatest source of error lies in the determination of the surface area of the bubble, A. In most cases (1,2) it was calculated from the photographed bubble projection and the assumption that the bubble has the shape of an oblate spheroid. This last assumption appears to be reasonably correct in the case of small bubbles of the size used in this work (d = 3 to 4 mm). However, it must be realized that the eccentricity of the bubble undergoes a constant variation during its rise in water and any area determination based on a single picture may be in error. This situation is complicated further by the fact that the changes of the area which the bubble undergoes take place in three dimensions and cannot be accurately represented by a two-dimensional picture.

To decrease this error and to obtain more reliable surface area data advantage was taken of the high speed of the camera which made it possible to take a great number of pictures during a short time interval. In the calculation, the surface area of the bubble was defined as that of an oblate spheroid of a volume equal to that of the bubble and having the minor semi-axis equal "b". The magnitude of "b" corresponding to a certain bubble volume was determined by averaging a number of its values in a region slightly above and below the volume in question. Taking as the time interval 1/10 of a second, corresponding to about 1 inch of the bubble path, about 100 pictures are available for the measurement. Usually about 5 to 10 representative bubble pictures were selected for the measurement of "b".

The surface area "A" corresponding to a given bubble volume "V" was then computed from the following relationship:

$$A = 1.50 \frac{V}{b} + 3.14 \frac{b^2}{\zeta_s} \log_e \frac{1 + \zeta_s}{1 - \zeta_s} \qquad (7)$$

where ζ_s is the eccentricity of the spheroid expressed in terms of its volume and the minor semi-axis:

$$\zeta_s = (1 - 4.19 \frac{b^3}{V})^{1/2}$$

Figure 7 shows the results of the three preliminary runs conducted with carbon dioxide bubbles of the same size under analogous experimental conditions. The change in volumes of the rising bubbles was plotted against the distance from the point of the common volume.

It can be seen that the spread of the experimental data is well within four percent of the original volume, which is a very satisfactory reproducibility considering the random behavior of the bubble.

Figure 8 is a graphical presentation of the results of one of these tests. Here the volume, surface area and height are plotted against time. The value of K_L calculated from this plot by means of eq. (6) was 0.0183 cm/sec. A similar calculation for the second run gave a somewhat higher result of 0.0199 cm/sec. The values of K_L for the third run could not be calculated because not enough bubble pictures were taken to estimate the surface areas.

FURTHER IMPROVEMENT OF THE BUBBLE RELEASE SYSTEM

It has been shown that the use of a hypodermic needle and a rubber membrane as a gland gave satisfactory results in the determination of the volume changes of a dissolving gas bubble. However, it was found that in some cases water could leak into the needle partially displacing the bubble and that variation in the time of release has an effect on the oscillation

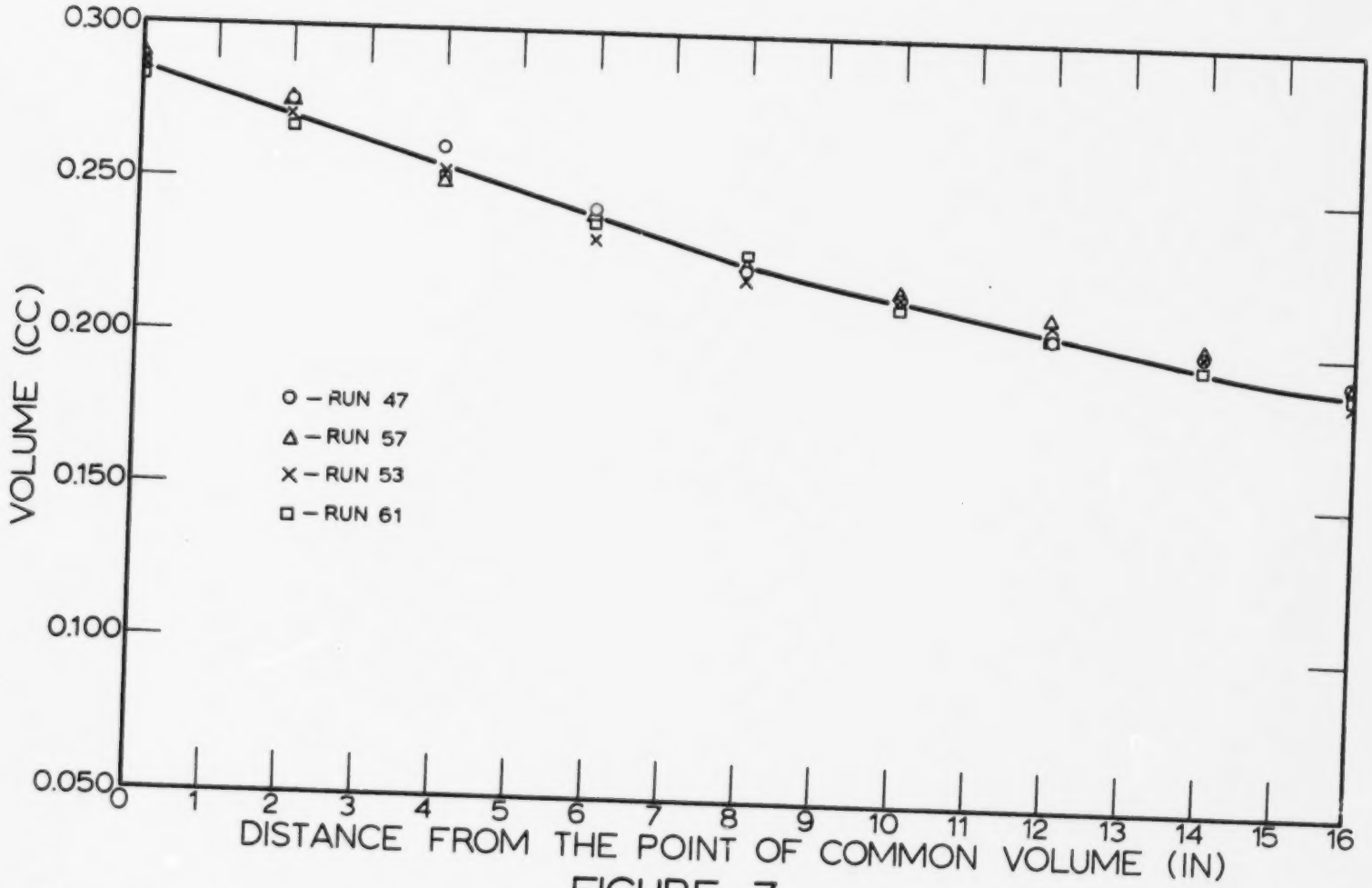

FIGURE 7

DISTANCE FROM THE POINT OF COMMON VOLUME vs VOLUME

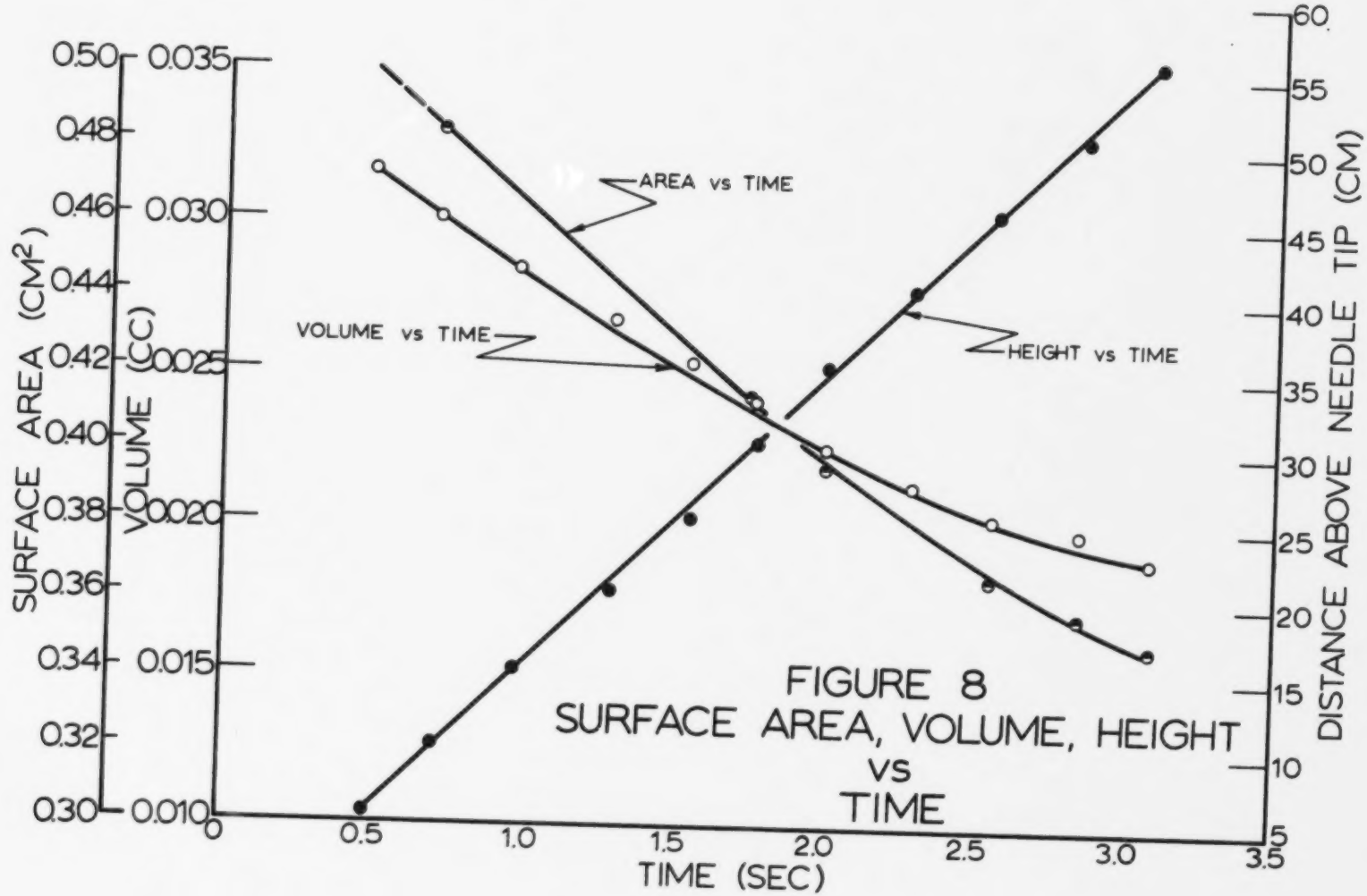

FIGURE 8
SURFACE AREA, VOLUME, HEIGHT
vs
TIME

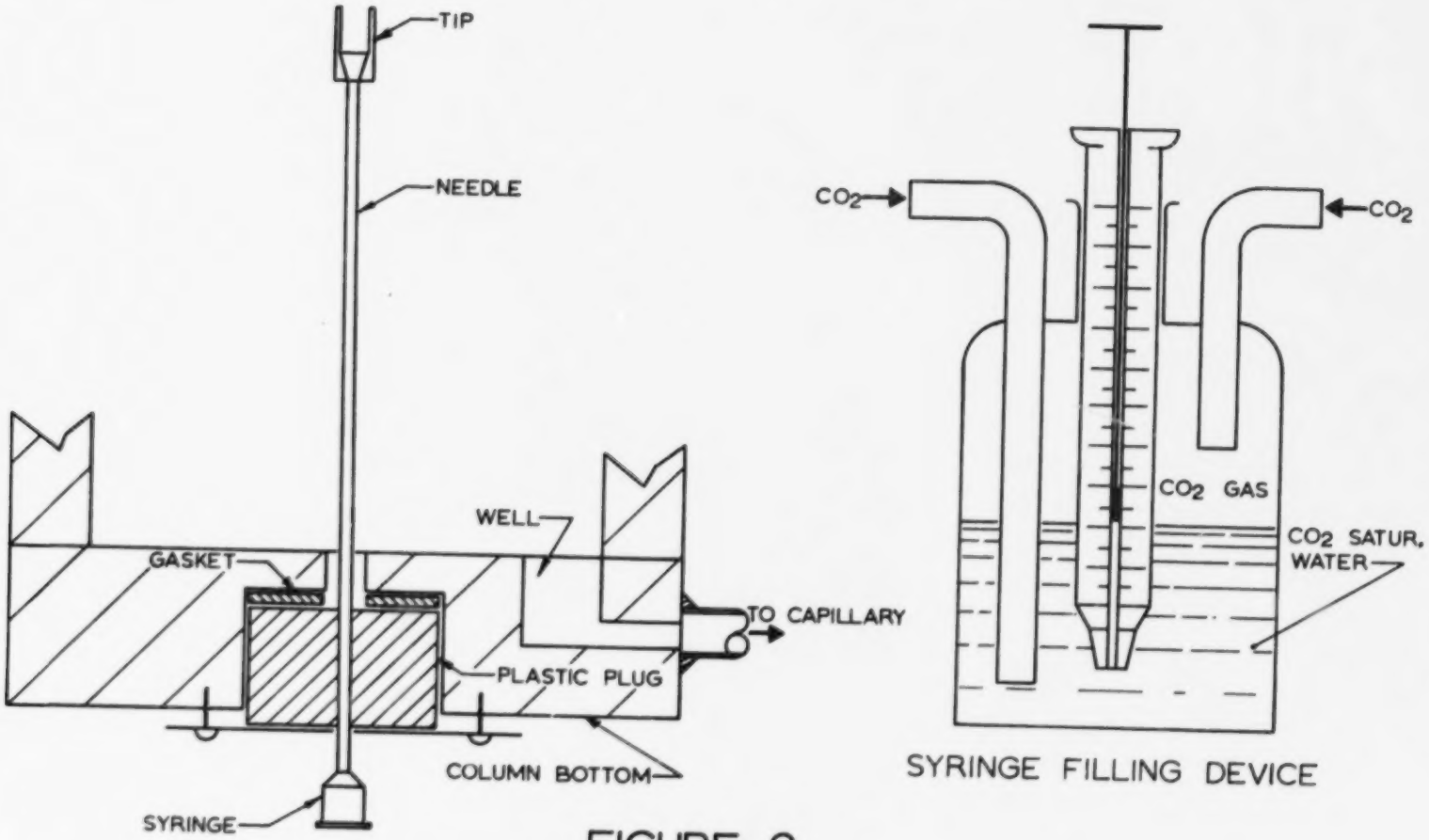

FIGURE 9
BUBBLE RELEASE ARRANGEMENT
(PERMANENT NEEDLE)

and path of the ascending bubble. These factors could result in errors. In order to decrease the possibility of these errors and to make the system more reliable it was decided to investigate a different bubble release arrangement which is shown in Figure 9. Here the needle is rigidly mounted in the base of the column. It is of a small diameter (I.D.: 0.020 inch) and is provided with a stainless steel tip. When the absorption column is filled, the needle is also filled with water until a small drop forms at the bottom of the needle. To introduce the gas bubble into the column the syringe is first filled (see Figure 9) with a saturated solution of the gas in the quantity equal to the volume of the needle and tip. After the solution has been introduced the syringe is raised above the level of the liquid and the required quantity of gas is sucked into it. In order to avoid dissolution of the gas in the water in the needle, the syringe is immersed again in the saturated solution and a small quantity of it (0.004 cm^3) is introduced. The filling of the syringe consists, therefore, of a small quantity of the saturated solution at the top, the plug of the gas, and again saturated solution of the gas at the bottom. After filling, the syringe is inserted in the hub of the needle. By pressing the plunger the saturated solution (0.004 cm^3) followed by the gas is introduced into the needle and the bubble is released when the plunger reaches the top of the syringe. To insure a uniform time of release the plunger of the syringe is actuated by a spring under constant tension.

The system as described above is presently under investigation. It is expected that it will improve the uniformity of bubble release and therefore the reproducibility of the system.

NOTATION

A = surface area of bubble: cm^2

a = major semi-axis of a spheroid: cm

b = minor semi-axis of a spheroid: cm

C_o = equilibrium concentration of the gas: g moles/cm^3

C_L = bulk concentration of the gas: g moles/cm^3

c' = conversion factor: $g/cm^2 = 0.9678 \times 10^{-3}$ atm

h = height of column measured from column base to bubble: cm

H = Henry's Law constant: g mole/cm^3 atm

K_L = over-all mass transfer coefficient: g moles/cm^2.sec(g moles/cm^3)

n = g moles of gas

P = pressure of gas inside bubble: atm

P_B = barometric pressure: atm

R = gas constant: 82.057 atm.cm^3/g mole, °K

T = °K

t = time, seconds

V = bubble volume, cm^3

δ = eccentricity: $(1 - \frac{b^2}{a^2})^{1/2}$ or $(1 - 4.19 \frac{b^3}{V})^{1/2}$

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